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INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(51) International Patent Classification 6 : C04B 16/02, 16/12	A1	(11) International Publication Number: **WO 97/18173** (43) International Publication Date: 22 May 1997 (22.05.97)

(21) International Application Number: PCT/US96/18363

(22) International Filing Date: 15 November 1996 (15.11.96)

(30) Priority Data:
08/558,847 15 November 1995 (15.11.95) US

(71)(72) Applicants and Inventors: SOROUSHIAN, Parviz [US/US]; DPD, Inc., 2000 Turner Street, Lansing, MI 48906 (US). HSU, Jer-Wen [CN/US]; 2000 Turner Street, Lansing, MI 48906 (US).

(74) Common Representative: SOROUSHIAN, Parviz; DPD, Inc., 2000 Turner Street, Lansing, MI 48906 (US).

(81) Designated States: AL, AM, AT, AU, AZ, BB, BG, BR, BY, CA, CH, CN, CZ, DE, DK, EE, ES, FI, GB, GE, HU, IL, IS, JP, KE, KG, KP, KR, KZ, LK, LR, LS, LT, LU, LV, MD, MG, MK, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, TJ, TM, TR, TT, UA, UG, US, UZ, VN, ARIPO patent (KE, LS, MW, SD, SZ, UG), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, CH, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, ML, MR, NE, SN, TD, TG).

Published
With international search report.

(54) Title: DISPERSION OF PLANT PULP IN CONCRETE AND USE THEREOF

(57) Abstract

The wet pulp derived from wood, plants or waste paper is dried to a relatively low density with limited bonding developed between fibers. The resulting dried pulp is conveniently broken up, using limited mechanical energy and without substantially damaging the fibers, into individual fibers for use as reinforcement in cement-based materials.

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Dispersion of Plant Pulp in Concrete and Use Thereof

Cross-References to Related Applications

None.

Statement as to Rights to Inventions Made Under Federally-Sponsored Research and Development

None.

Background of the Invention

Field of the Invention

The invention relates to plant and wood pulp fibers and cement-based materials, and especially to the processing of plant and wood pulp fibers for the reinforcement of cement-based materials.

Description of the Prior Art

Pulp fibers derived by mechanical, thermo-mechanical and chemical methods, or combinations thereof, from different wood species and plants offer excellent characteristics for the reinforcement of cement-based materials. These characteristics include:

(1) Wood and plant pulp fibers provide equivalent cross-sectional diameters of about 1-100 microns, and lengths of about 0.2-10 mm. Equivalent diameter here refers to the diameter of a circle which provides the same cross-sectional area as the fiber. The fine diameter of such fibers increases their surface area and the number of fibers per unit weight. For example, 1 kg of southern pine kraft pulp fibers provides close to 1.8

billion fibers with total surface area of approximately 250 million mm^2; plant and wood pulp fibers thus provide a close fiber spacing and a high fiber surface area per unit weight addition to cement-based materials. These characteristics benefit the action of plant and wood pulp fibers as reinforcement in cement-based matrices. Furthermore, the relatively high length-to-equivalent diameter ratio of plant and wood pulp fibers provides for desirable anchorage of fibers within cement-based matrices and helps fully mobilize the tensile strength of fibers in the composite system. The equivalent diameter of many plant and pulp fibers is within the range of the particle size of Portland cement; this favors compact packing of the cement particles around fibers, and favors improvements in the hardened material micro-structure and properties.

(2) Plant and wood pulp fibers generally provide high levels of tensile strength and elastic modulus, which benefit their effectiveness as reinforcement in cement-based matrices.

(3) Plant and wood pulp fibers provide hydrophilic surfaces which also develop strong bonding to cement-based matrices. Hydrophilic fiber surfaces facilitate uniform dispersion of fibers in the aqueous environment of fresh cement-based matrices. The strong bonding of these surfaces to the cement-based matrix also favors the effectiveness of plant and wood pulp fibers as discrete reinforcement in cement-based materials.

(4) Many plant and wood pulp fibers, especially kraft wood pulp, are stable in alkaline environments. The environment in many cement-based matrices is alkline and thus the alkali resistance of fibers favors improved long-term durability of fiber reinforced composite systems.

The pulping process for deriving fibers from wood and plant, or from waste paper incorporating such fibers, is a wet process and yields fibers with a moisture content in excess of about 90% by weight. If the pulp is to be marketed for the production of paper products, its moisture content is reduced through pressing and heating. Since pressing presents a more efficient approach to moisture removal, pulp mills rely heavily on pressing, in lieu of heating, to dry the wet pulp into compacted sheets of pulp with a high density of about 0.85-1.2 g/cm^3 at a moisture content of less than about 10% by weight. Heavy use of pressing in the drying process leads to the development of strong hydrogen bonds between a substantial fraction of the surfaces of adjacent fibers. Any subsequent use of the pulp as individual fibers involves breaking of the hydrogen bonds between fibers to separate them from each other. This process of separating pulp fibers from each other should not damage or cut the fibers. Noting that moisture helps break the hydrogen bonds, wetting of the pulp to moisture contents exceeding about 90% by weight is used in paper production to facilitate the separation of individual fibers by mechanical action.

The processing of plant and wood pulp fibers into fiber reinforced cement composites has traditionally involved the use of the available paper pulp which has been dried by heavy reliance on pressing accompanied with heating into a dense pulp with strong fiber-to-fiber bonding. The common method for separating individual fibers from such highly pressed pulp sheets involves the use of moisture together with mechanical action to break the fiber bonds and make individual fibers available for uniform dispersion in cement-based matrices. Subsequently, when excess water is used in the fiber separation and dispersion process, a fraction of the water may be removed from the wet composite system using vacuum and mechanical pressure. This process has been used, for example,

in U.S. Patent No. 4,985,119 to Vinson et al. (1991) and U.S. Patent No. 5,102,596 to Lempfer et al. (1992). The strong fiber-to-fiber bonding in such pulp sheets, which have been dried through heavy pressing for use in paper production, increases the need for moisture and mechanical action to break up the pulp into individual fibers for use as reinforcement in cement-based matrices. The resistance provided by the strong fiber-to-fiber bonds increases the damage to fibers in the process, which is a detriment to the reinforcement efficiency of fibers. This strong bond also renders the mechanical separation action less effective and thus leaves agglomerates of multiple unseparated fibers.

The use of mechanical action without added moisture is also an option for separating the fibers from the highly pressed pulp sheets, or even from paper products where strong hydrogen bonding between fibers provides for the integrity of paper. Dry separation of pulp fibers has been referred to in U.S. Patent No. 3,753,749 to Nutt (1973). The dry process of separating bonded pulp fibers does not have the benefit of water to break hydrogen bonds between fibers and thus relies on a more intense mechanical action to break up the highly pressed pulp sheets into individual fibers for use as discrete reinforcement in cement-based matrices. Such mechanical action may be applied to the pulp sheets using a mill such as hammer mill, pin mill, or the like. The intense mechanical action required to break up the highly pressed pulp sheets causes increased damage to fibers and leaves many broken fibers and fines with reduced reinforcement efficiency. Also, given the strong hydrogen bonding between fiber surfaces in a highly pressed pulp sheet, dry mechanical processing still leaves a fraction of the pulp mass as agglomerates or knots comprising multiple fibers which are still bonded together. Such multiple fiber

agglomerates are not effective as reinforcement in cement-based matrices and could actually damage the performance of such matrices.

It is, therefore, the principal object of the present invention to provide an improved method of drying pulp which facilitates subsequent separation of the dried pulp into individual fibers for use as discrete reinforcement in cement-based materials.

It is also an object of the invention to provide improved cellulose fiber reinforced cement-based materials with desirable performance in fresh and hardened states.

Summary of the Invention

The principal object of the present invention is to dry wood and plant pulp fibers so that fiber-to-fiber bonding is reduced, and thus the dried pulp can be effectively and efficiently separated into individual fibers for addition to cement-based materials as discrete reinforcement. The drying process differs from that used in paper pulp production by avoiding heavy pressing of the pulp in the drying process, or the addition of surfactant to the wet pulp prior to drying, or a combination thereof. The reduced fiber-to-fiber bonding in such dried pulp facilitates subsequent processing of the pulp into individual fibers using mechanical action with or without the use of water in the process. The mechanical energy required to break up the dried pulp into individual fibers is thus reduced, and the separation process causes less damage to fibers and leaves less multiple fiber agglomerates. The resulting fibers are more effective as discrete reinforcement in cement-based materials.

Brief Description of the Drawings

None.

Description of the Preferred Embodiments

The addition of slender fibers to cement-based materials improves the cohesiveness, finishability, pumpability, segregation resistance and cracking resistance of such mixtures in the fresh state, and also enhances their toughness, impact resistance, strength, cracking resistance and durability in the hardened state. To be effective, individual fibers should be uniformly dispersed in cement-based materials. Most fibers, including steel and nylon fibers, are produced as individual fibers which do not have a tendency for fiber-to-fiber bonding. The resulting fibers can thus be added to fresh cement-based mixtures as individual fibers to be dispersed in the mix through the mechanical mixing action. Cellulose fibers are derived from different softwood and hardwood species, plants such as flax and cotton, or waste paper in a wet process which may involve thermal, mechanical and chemical effects or combinations thereof. The resulting wet pulp fibers contain more than 90% by weight moisture content, and should be dried to a moisture content less than 10% by weight prior to shipment. The common drying process relies heavily on pressing which is more efficient than heating for the drying of pulp; this process produces strong hydrogen bonds between fibers and produces pulp sheets with a relatively high density of 0.85-1.2 g/cm^3. These hydrogen bonds should be subsequently broken in order to produce individual fibers needed for the reinforcement of cement-based materials. The breaking of fiber-to-fiber bonds in dried pulp can be accomplished using mechanical action with or without the addition of moisture to the pulp. The use of moisture weakens fiber-to-fiber bonding and thus facilitates the separation of fibers by mechanical action. When water is added to the previously dried pulp, the moistened pulp with about 50-1000% moisture content is subjected to blending,

beating or milling actions, or the like, for breaking up the pulp and reducing it to individual fibers. Without the addition of moisture, one may use the mechanical action of a mill such as a pin mill, a hammer mill, or the like in order to overcome the fiber-to-fiber bonds and separate individual fibers from the dried pulp. The strong hydrogen bonding between cellulose fiber surfaces in the pulp which has been dried with heavy reliance on pressing, as is the case in the pulp marketed to the paper industry, necessitates the use of a more intense mechanical action for the separation of individual fibers from the pulp. This not only increases the energy requirement in the process of deriving individual fibers from the dried pulp, but also causes damage to fibers, breaks many fibers into short fibers and fines, and leaves many multiple fiber agglomerates. The damaged and broken fibers as well as multiple fiber agglomerates are not effective as discrete reinforcement in cement-based materials. This is a detriment to the reinforcement efficiency of fibers separated from the highly pressed pulp dried for marketing to the paper industry when such fibers are used as reinforcement in cement-based materials. It is thus desirable to refine the drying process that is commonly applied to paper pulp in order to reduce the extent of fiber-to-fiber bonding in the dried pulp and thus enhance the effectiveness and efficiency of the subsequent process of breaking up of the dried pulp into individual fibers for use as discrete reinforcement in cement-based materials.

The invention described herein refines the drying process of wet pulp fibers to achieve moisture contents below about 10% by weight through reducing the reliance on pressing and increasing the reliance on heating in the drying process, addition of surfactant to the wet pulp prior to drying, or a combination thereof. The result is a lighter-weight or fluff pulp that is less dense when compared with paper pulp, with a density of about 0.2-

0.8 g/cm^3; the fiber surfaces in such dried pulp develop less hydrogen bonding to each other. The pulp can thus be broken up into individual fibers more effectively and efficiently with or without the addition of water. Less mechanical energy is needed in separating individual fibers from such dried pulp and the process causes less damage to fibers and leaves less broken fibers, fines, or multiple fiber agglomerates. The resulting individual fibers thus provide a higher reinforcement efficiency when dispersed in cement-based matrices.

The dried pulp thus obtained by less reliance on pressing in the drying process, addition of surfactant prior to or during drying, or a combination thereof, is then broken up into individual fibers by mechanical action with or without the addition of water to the pulp. When moisture is not added to the dried pulp, the mechanical action to separate individual fibers from the dried pulp may be provided by a mill, such as hammer mill, pin mill, or the like. When moisture is added, the wet pulp reaches moisture contents of about 50-1000% by weight, with the added moisture being part or all of the water needed in the cement-based mix, or even exceeding that needed in the mix. The excess moisture, if any, should be subsequently removed from the mix using vacuum, pressure, heat, or combinations thereof. The separation process of such pulp dried to reduce fiber-to-fiber bonding, with or without the addition of water, consumes less energy, causes less damage to fibers, and leaves less broken fibers, fines, or multiple fiber agglomerates. The result is individualized fibers which can be conveniently dispersed in cement-based mixtures and offer high levels of reinforcement efficiency. The fibers separated from such pulp may be added to cement-based mixtures at about 0.01-40% volume fraction, and preferably at about 0.05-5% volume fraction which corresponds to approximately 0.7-45

kg/m^3. Such cement-based mixtures may be concrete comprising cement, fine aggregate, coarse aggregate and different admixtures. Fine and coarse aggregates could be of mineral, synthetic, metallic or organic sources with fine aggregate-to-cement weight ratios of about 0.1-30 and coarse aggregate-to-cement weight ratios of about 0.1-30. The maximum particle size in fine and coarse aggregates is less than about 6 mm and less than about 75 mm in size, respectively. The cement binder in concrete may be hydraulic cement such as different types of Portland cement, blended cement, expansive cement, high-alumina cement, masonry cement, block cement, magnesium phosphate cement, or set-regulated cement. The water-cement ratio in concrete is about 0.1-0.9 by weight. Various admixtures may also be used in concrete including air-entraining admixtures, set-accelerating admixtures, set-retarding admixtures, polymeric admixtures, pozzolanic admixtures, water-reducers, superplasticizers, or combinations thereof. The cement-based mixtures into which the cellulose fibers are dispersed may also be mortar which is essentially the same as concrete but without the coarse aggregate. The maximum particle size of fine aggregates in mortar could be less than 5 mm, and as small as 0.05 mm. The cement binder in mortar could be any of the hydraulic cements used in concrete; this binder could also be of hydratable type such as gypsum or plaster. The water-cement weight ratio during processing could exceed the 0.1-0.9 range given above for concrete; special processing techniques which involve the addition of water to pulp during breaking up of the dried pulp may yield water-cement ratios of about 100 during processing; the excess water would be removed using vacuum, pressure, heat, or combinations thereof in order to reduce the water-cement ratio of the end product to about 0.1-0.9 by weight.

Cellulose fiber reinforced mortar and concrete mixtures can be mixed, transported, placed, pumped, sprayed, slipformed, extruded, consolidated, finished and cured into end products using all techniques applicable to mortar and concrete. The fibers could also be added at any step during the mixing process. Alternatively, some or all the mix ingredients could be added to the pulp prior to or during the breaking down of the dried pulp into individual fibers, with the remaining mix ingredients, if any, added later during the mixing process.

The presence of cellulose fibers in the mix increases the cohesiveness and segregation resistance of the fresh mix and thus benefits some steps in the processing of mortar and concrete; such steps include pumping, spraying, extrusion, finishing and the like. As understood from the following Examples, the reinforcing action of cellulose fibers in mortar and concrete enhances their toughness, strength, impact resistance, cracking resistance, fatigue life, and durability.

Example 1

Pulp sheets dried by a conventional process and a process according to the invention, respectively, were broken up into individual fibers using a hammer mill. The same wet pulp was used to produce these pulp sheets; this wet pulp was obtained by the kraft chemical pulping of southern pine and was also bleached. The first sheet was dried by the common method applied to paper pulp where pressing of the wet pulp is heavily relied on for the removal of water, yielding an average density of 0.86 g/cm^3. The second sheet according to the invention was dried with less reliance on pressing and more on heating in order to reduce the density of the dried pulp sheet to 0.64 g/cm^3. Separation of individual fibers from the two dried pulp sheets in the same hammer mill yielded the

following results. The conventionally dried pulp which had strong hydrogen bonding between fibers produced 4% by weight of multiple fiber agglomerates, 19% by weight of fines passing #200 (74 micron opening) sieve, and an average fiber length of 0.92 mm. The pulp dried according to the invention with less reliance on pressing which had reduced fiber-to-fiber hydrogen bonding produced 0,1% by weight of multiple fiber agglomerates, 5.5% by weight of fines passing #200 (74 micron opening) sieve, and an average fiber length of 1.3 mm. The drying process of the invention which reduced fiber-to-fiber hydrogen bonding was thus successful in enhancing the effectiveness of the hammer mill in separating the pulp sheets into individual fibers, and also in reducing the breaking of fibers in the hammer mill.

The individual fibers separated from the two pulp sheets as above were added to two similar fresh concrete mixtures at a dosage of 0.9 kg/m^3 which corresponds to a fiber volume fraction of 0.06%. The concrete mix comprised Type I Portland cement, water, crushed limestone of 19 mm maximum particle size as coarse aggregate and natural sand of 5 mm maximum particle size as fine aggregate. The water-cement, fine aggregate-cement and coarse aggregate-cement weight ratios were 0.564, 2.17 and 3.43, respectively. The fibers were added to the fresh mix after all other ingredients were mixed in a rotary drum mixer. Mixing was continued for about 3 minutes after the addition of fibers in order to achieve a uniform dispersion of fibers. The fresh mix was then placed in molds and vibrated into prismatic specimens of 100 mm square cross sections and a length of 356 mm. These specimens were moist cured for 48 hours and then tested in flexure by four-point loading over a span of 300 mm. The flexural strength obtained with fibers separated from conventionally dried paper pulp with strong hydrogen bonding was 4.9

MPa while that with fibers separated according to the invention with less fiber-to-fiber bonding was 6.2 MPa. This confirms that cellulose fibers from the same source offer higher levels of reinforcement efficiency when separated from a dried pulp of lower density with less extensive fiber-to-fiber hydrogen bonding. Analysis of the fractured surfaces of the flexural specimens yielded about 10% lower variation in fiber count per unit area for the invention example versus the comparative example; this indicates that pulp fibers dried and separated according to the invention can be dispersed more uniformly in concrete.

Example 2

The cellulose fiber reinforced concrete mixture according to the invention as in Example 1 above as well as a corresponding fresh mix without the addition of fibers, were molded and consolidated into specimens for the performance of compression (ASTM C 39), drop-ball impact (ACI Committee 544) and fracture bend (RILEM TC89-FMT) tests. The compression specimens were 100 mm in diameter and 200 mm high. The impact test specimens were 150 mm in diameter and 64 mm mm high. The fracture test specimens were 100x100x457 mm prisms. Three compression, three impact, and three fracture test specimens were prepared from each mix. The specimens were kept inside their molds under a wet burlap for 24 hours, and were then demolded and subjected to 14 days of moist curing followed by 14 days of air drying in laboratory prior to testing. The plain and cellulose fiber reinforced concretes produced average compressive strengths of 30 and 40 MPa, respectively, ultimate impact strengths of 19 and 99 drops, respectively, and fracture strengths of 3.1 and 5.3 MPa, respectively. These results confirm the effectiveness of the

cellulose fibers derived according to the invention in enhancing the impact resistance and strength characteristics of concrete.

Example 3

The cellulose fiber derived according to the invention in Example 1 above was added to a mortar mixture at 1% volume fraction. The mortar mix comprised Type 1 Portland cement, silica fume, silica sand of 1.2 mm maximum particle size, water, and superplasticizer. The silica sand-to-cement weight ratio was 1.075, silica fume-to-cement weight ratio was 0.08, and water-to-cement weight ratio was 0.37. The dosage of superplasticizer was 5.1 l/m^3. The mortar mix was first prepared in a conventional mortar mixer, and fibers were then added and dispersed in the mix through continuation of mixing for about 3 minutes. The fresh mortar mix was molded and vibrated into twelve prismatic specimens which were 25 mm thick and 50 mm wide. Comparative specimens were also made from plain mortar of the same components without the addition of cellulose fibers. All the specimens were retained in their molds under wet burlap for 24 hours, and were subsequently demolded, moist-cured for 14 days, and air-dried for another 14 days. Three of the specimens from each mix were then subjected to four-point flexure testing on a span of 150 mm. The remaining specimens were divided to groups of three from each mix and each group subjected to one of the following accelerated aging conditions: (1) 25 cycles of wetting and drying, with each cycle comprising 3 hours of water spray at 23 degrees C and 3 hours of drying at 60 degrees C; and (2) 14 days of immersion in warm water at 60 degrees C. All the specimens were subsequently subjected to four-point flexure testing on a span of 150 mm. The unaged and aged flexural strength test results are described below.

The plain and cellulose fiber reinforced mortars reached unaged flexural strengths at 28 days of 4 MPa and 7.7 MPa, respectively. This confirms the effectiveness of cellulose fibers in enhancing the flexural strength of mortar. After both accelerated aging processes, the plain and fiber reinforced mortars retained more than 95% of their flexural strength. Hence, cellulose fibers retained their high flexural strength even after aging.

Claims

We claim:

1. A process for making a cellulose fiber reinforced cement-based material using cellulose fibers derived through a wet process using at least one of mechanical, thermal and chemical pulping methods, said process comprising the steps of:
   a. drying wet cellulose pulp to a density of about 0.2-0.8 g/cm^3 and moisture content of about 0.1%-10% by weight;
   b. separating the dried pulp into individual fibers through breaking fiber-to-fiber bonds using mechanical action; and
   c. adding the separated individual fibers at about 0.01%-40% fiber volume fraction to a cement-based material comprising cement, aggregates, water, with fibers and other ingredients added in any sequence, at an aggregate-to-cement weight ratio of about 0.1-60 and water-to-cement weight ratio of about 0.1-100, where the cement is any hydraulic or hydratable cement, said aggregates are of at least one of mineral, synthetic, metallic and organic sources with about 0.05-5 mm minimum particle size and about 0.1-100 mm maximum aggregate particle size, with the fiber and other ingredients combined into a homogeneous blend.

2. The process of Claim 1, wherein the cement-based material is concrete, said minimum aggregate particle size is about 0.05-3 mm, said maximum aggregate particle size is about 9-100 mm, said aggregate-to-cement weight ratio is about 1-10, and said water-to-cement weight ratio is about 0.1-0.9, said separation step involves separating the individual fibers from the dried pulp without the addition of

water, and said separated fibers are added to said concrete at about 0.01%-5% volume fraction.

3. The process of Claim 1, wherein the cement-based material is mortar, said minimum aggregate particle size is about 0.05-1 mm, said maximum aggregate particle size is about 0.1-6 mm, said aggregate-to-cement weight ratio is about 0.1-6, said water-to-cement weight ratio is about 0.1-0.9, said separation step involves separating said individual fibers from the dried pulp without the addition of water, and said individual fibers are added to the mortar at about 0.05%-20% volume fraction.

4. The process of Claim 1 wherein, the said separation step involves separating the inidividual fibers from the dried pulp using a mill without the addition of water and the process further comprises the step of compacting the separated individual fibers to a density of about 0.01-1 g/cm^3 at about 0.1%-10% moisture content by weight prior to said addition step.

5. The process of Claim 1, wherein the fiber separation step is accomplished with some or all the ingredients of cement-based material added to the pulp.

6. The process of Claim 1, comprising a further step of forming the cement-based material into a fiber reinforced cement-based structure, said forming step including incorporation of about 0.01%-30% volume fraction of at least one of continuous and discrete reinforcement of steel, synthetic, glass, mineral, natural and fiber reinforced polymer and metal matrix composite types into the fiber reinforced cement-based structure.

7. The process of Claim 1, wherein said cement-based material further comprises admixtures of at least one of chemical, mineral, polymeric and air-entraining types.

8. A hardened cellulose fiber reinforced cement structure comprising:

a. about 0.01%-40% by volume of cellulose fibers separated from pulp which has been derived from at least one of wood, plants, and waste paper in a wet process and dried to a density of about 0.2-0.8 g/cm^3 and moisture content of about 0.1%-10% by weight; and

b. a cement-based binder comprising cement, aggregates and water, at an aggregate-to-cement weight ratio of about 0.1-60 and a water-to-cement weight ratio of about 0.1-100, where cement is any hydraulic or hydratable cement, said aggregate is of at least one of mineral, synthetic, metallic and organic sources, with about 0.05-5 mm minimum aggregate particle size and about 0.1-100 mm maximum aggregate particle size.

9. The cellulose fiber reinforced structure of Claim 8, wherein the cement-based binder is concrete, said minimum aggregate particle size is about 0.05-3 mm, said maximum aggregate particle size is about 9-100 mm, said aggregate-to-cement weight ratio is about 1-10 and said water-to-cement weight ratio is about 0.1-0.9, said cellulose fibers are separated from the dried pulp without the addition of water, and added to the concrete at about 0.01%-5% volume fraction.

10. The cellulose fiber reinforced structure of Claim 8, wherein the cement-based binder is mortar, said minimum aggregate particle size is about 0.05-1 mm, said maximum aggregate particle size is about 0.1-6 mm, said aggregate-to-cement

weight ratio is about 0.1-6, said water-to-cement weight ratio is about 0.1-0.9, and said cellulose fibers are separated from the dried pulp without the addition of water, and added to the mortar at about 0.05%-20% volume fraction.

11. The cellulose fiber reinforced structure of Claim 8, wherein said structure further comprises about 0.01%-30% volume fraction of at least one of continuous and discrete reinforcement of at least one of steel, synthetic, glass, mineral, natural and fiber reinforced polymer and metal matrix composite types.

12. The cellulose fiber reinforced structure of Claim 8, wherein said cement-based binder further comprises admixtures of at least one of chemical, mineral, polymeric and air-entraining types.

INTERNATIONAL SEARCH REPORT

International application No.
PCT/US96/18363

A. CLASSIFICATION OF SUBJECT MATTER

IPC(6) :C04B 16/02, 16/12
US CL :106/731, 805
According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

U.S. : 106/731, 805

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched
NONE

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)
NONE

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	US 3,753,749 A [NUTT] 21 August 1973, col. 3, lines 45-66.	1-12
A	US 4,985,119 A [VINSON et al] 15 January 1991, see entire document.	1-12
A	US 5,102,596 A [LEMPFER et al] 07 April 1992, see entire document.	1-12

☐ Further documents are listed in the continuation of Box C. ☐ See patent family annex.

* Special categories of cited documents:
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Washington, D.C. 20231
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Authorized officer
for DAVID M BRUNSMAN
Telephone No. (703) 308-0661